

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re: Kendall, et al. Docket No.: IR-2795(EC)
Serial No.: 09/888,793 Filing Date: 6/25/2001
Examiner: Robert D. Harlan Art Unit: 1713
For: "Metathesis Polymerization Adhesives and Coatings"

#9
Jeddy
12/18/02

Nov. 22, 2002

Assistant Commissioner for Patents
Washington, DC 20231

DECLARATION PER 37 C.F.R. 1.131

Sir:

This is in response to the Office Action dated May 22, 2002. The undersigned
Applicants declare the following:

As a below named inventor, we hereby declare that:

Our residence, post office address and citizenship are as stated below next
to our names; and

we believe we are the original, first and joint inventors of the subject
matter which is claimed and for which a patent is sought on the invention entitled,
"Metathesis Polymerization Adhesives and Coatings" the specification of which was filed
6-25-01 in the United States Patent and Trademark Office.

Submitted as attached is objective evidence of invention by Applicants prior to the
reference effective date, December 5, 2000 of Japanese Kokai Patent Application 336320
as attached photocopy pages of Notebooks and is evidence of actual reduction to
practice, summarized as follows:

RECEIVED
DEC 02 2002
TC 1700

Pages 28 and 29 from Notebook No. 8148, evidencing that polymerization between rubber and metal with bonding was achieved using Grubb's catalyst and ENB monomer.

Pages 58 and 59 from Notebook No. 8060 showing that Grubbs catalyst coatings on EPDM substrate followed by coating of p-ENB resulted in a polymerized coating; and catalyst coatings on glass slides followed by spraying of ENB monomer resulted in a peelable solid coating film.

Page 57 from Notebook No. 8148 showing that monomer mixtures of ENB and TCHP polymerized with Grubb's catalyst.

Pages 50 and 51 of Notebook No. 8297 demonstrated contact metathesis adhesion between different polyolefin (LDPE) substrate bonded to itself with 5,5-bis(chloromethyl)-2-norbornene using Grubb's catalyst.

Pages 41 and 42 of Notebook No. 8327 demonstrated the bonding of polypropylene to itself using 5,6-bis(chloromethyl)-2-norbornene delivered from a 2-part cartridge.

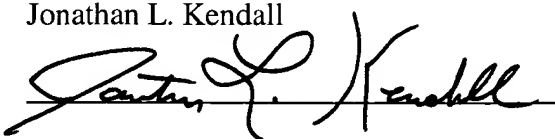
Pages 66 and 67 of Notebook No. 8327 demonstrated bonding of polypropylene using ENB and Grubb's catalyst.


Pages 64 to 65 of Notebook No. 8363 demonstrated formulation as a two part adhesive of ENB, NBD, silica, and an elastomer (Europrene) provided a measured amount of bond strength in lap shear samples.

Pages 11, 12, 15, 22 and 23 of Notebook No. 8374 demonstrated formulation as a two part adhesive of norbornadiene, ENB, Blendex, Cab-o-sil and Zeespheres provided a measured bond strength in lap shear polypropylene samples.

Pages 51 to 56, 59-60 and 89-90 of Notebook No. 8374 demonstrated 2-part adhesive formulations applied to bond polypropylene substrates.

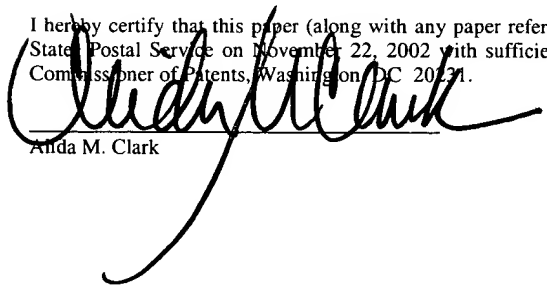
We further declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of first inventor: Jonathan L. Kendall
Inventor's Signature: 
Date: Nov. 22, 2002
Residence: Apex, North Carolina
Citizenship: United States of America
Post Office Address: 704 Nottingham Walk
Apex, North Carolina 27502

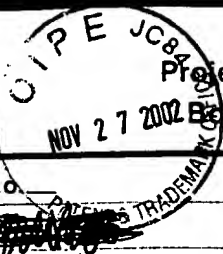
Full name of second inventor:
Inventor's Signature: 
Date: Nov. 22, 2002
Residence: Apex, North Carolina
Citizenship: United States of America
Post Office Address: 109 Lewey Brook Drive
Cary, North Carolina 27519

CERTIFICATE OF MAILING (37 CFR 1.8(A))

I hereby certify that this paper (along with any paper referred to as being attached or enclosed) is being deposited with the United States Postal Service on November 22, 2002 with sufficient postage as first class mail in an envelope addressed to the Assistant Commissioner of Patents, Washington, DC 20231.


Linda M. Clark


Date 11/22/02



Project No.

Book No. 8148

TITLE

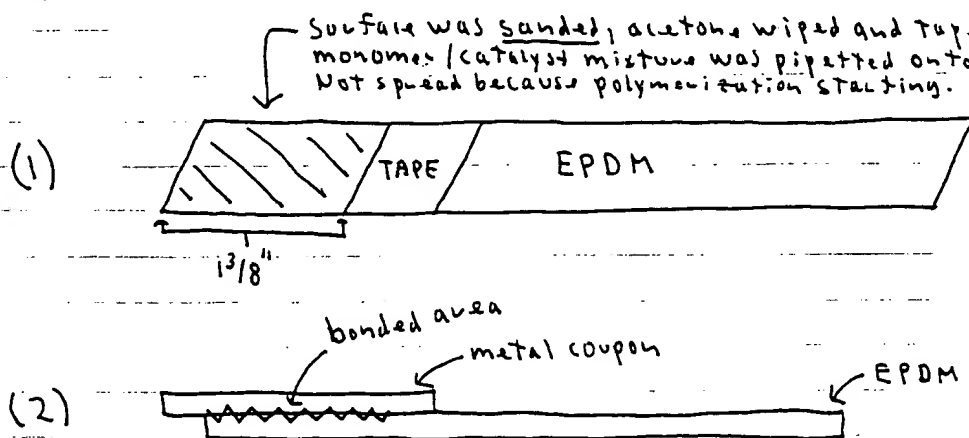
From Page No.

Rubber to Metal Bonding Using Monomer / Catalyst Mixture

Materials: ENB (CK8148-15-1); Grubbs's Catalyst (Strem); EPDM (J. Taylor); Grit Blasted Metal coupons (M. Righettini)

Procedure:

- 1) 0.0009g of catalyst (0.000001 moles) was weighed into a glass vial.
- 2) 2.8ml (2.5004g or 0.0208 moles) of ENB was quickly syringed into the glass vial and immediately stirred for a few seconds.
- 3) The monomer / catalyst mixture was then dropped onto the EPDM strip using a glass pipet. The metal coupon was finally placed over the treated EPDM surface and held in place until bonding occurred. A brown jar was placed over the bonded area. Monomer to catalyst molar ratio was 20,800:1.
- 4) Bonding procedure was as follows (pictured from above description):



Immediately, the acetone washed metal coupon was placed on the EPDM strip in treated area. Material came out the sides and polymerized during bonding. The coupon was held in place until hard to move.

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To Page No. 29

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

Chris Keck

From Page No. 28

5) 5 hrs after bonding, metal easily pulled apart from rubber. Nothing on metal. Rubber had a thin, elastic film stuck to it.

PART II

Steps 1-4 (pages 28) were repeated. However, this time the monomer/catalyst mixture was pipetted onto the metal surface and the EPDM quickly placed onto the metal surface. The reverse of the first experiment. 17 hrs after bonding, metal again easily pulled apart from the rubber. Like above, nothing on metal while rubber had a thin, elastic film stuck to it.

C.K.

~~XXXXXXXXXX~~

To Page No. _____

Witnessed & Understood by me,

S. J. [Signature]

Date

~~XXXXXXXXXX~~

Invented by

R c rded by *Chris Keck*

Date

~~XXXXXXXXXX~~

From Page N 58

Purpose: To prepare samples for surface analysis on CMP technology.
Ed Tokas will be taking different samples to NJ w/ MAS to conduct ESCA analysis.

Instructions: from Ed Tokas

Printed By: Ed Tokas

Page: 1

From: Ed Tokas (Approved)

To: Chris Kock, Russell Walls

CC: Ken Carter, Steve Rowe, Marlene Righettini, Lynn Yanyo

REGARDING

CMP Samples for Analysis

Chris and Russ,

Giving you a reminder that next week I will be going to conduct two separate surface analyses on CMP samples:

1st Tuesday Marlene and I will go to NCSU to run a cross sectional analysis on R/R bonded samples (one freshly prepared and one aged sample).

2nd On Wednesday I will be going to NJ w/ MAS to conduct ESCA analysis and will need the following samples:

Labeled as
Notebook Page

Sample

✓ -1

EPDM

(for all preps in this series use the 1/16 in. pads located in my office)

✓

-2

EPDM w/ catalyst

-3

EPDM w/ a thin layer of poly-ENB
(thinnest of three samples)

-4

EPDM w/ intermediate thickness of poly-ENB

-5

EPDM w/ thick layer of poly-ENB

-6 and -7

A free film of poly-ENB
(prepared on glass and carefully removed)

-8

A bulk polymerized poly-ENB
(prepare in a tube)

✓

-9

Metal coupon
(the grit blasted Fe coupons we have been using)

✓

-10

Metal w/ catalyst
(the grit blasted Fe coupons we have been using)

See me Monday AM to discuss.

Thanks Ed

Russell
Walls
~~Ed Tokas~~

Procedure/Log

Time

Comments

8:30 am

(~~10:00~~) Discussions with Ed Tokas to make clear what is to be done.

Since the samples are to be sprayed, I was instructed to prepare most of the samples. Chris Kock was instructed to make the bulk polymerized poly-ENB in a small test tube.

1:00 pm

Ed Tokas wanted to be present for first spraying.

0.75g of Grubbs catalyst was dissolved in 10ml MeCl₂ and swirled till dissolved.

1:10 pm

Coated 1/16" 6"x6" EPDM with catalyst on two passes, dry, and two more passes for a total of 4 passes. Ed said it looked fine. Asked that we wait 30 minutes before applying monomers.

wiped with acetone to clean first.

To Page No. 59

Witness d & Understood by m ,

James B. Weger

Date

~~11/11/93~~
11/11/93

Invented by

Recorded by

Russell Walls

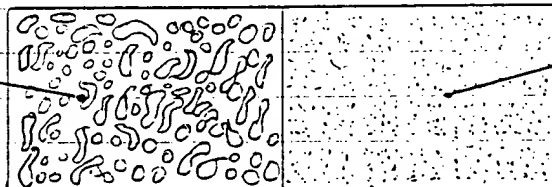
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~~11/11/93~~
11/11/93

TITLE ESCA Samples continuedFrom Page No. 58

- Time Comments
- 1:40 pm • Applied thin coat of poly-ENB by making two passes over the rubber surface. Allowed to dry in hood. Smell of monomer still present. 4 sample with just catalyst stored.
- 1:45 pm • Acetone wiped EPDM labelled as RW8060-58-1 with silver pen on back and placed in ziplock bag. Bag labelled and brown paper placed in front of surface to be tested so that it would not come in contact with plastic of bag.
- EPDM with just catalyst labelled as RW8060-58-2 and stored in ziplock bag just like RW8060-58-1.
- 2:00 pm • Forgot to note that at 1:40 pm I also coated two small metal coupons (not grit blasted) with 1 mils catalyst. Stored and labelled as RW8060-58-10 and placed in ziplock bag. The unsprayed coupons just wiped with acetone stored and labelled as RW8060-58-9.
- Also at 1:40 pm I coated one micro glass slide with catalyst.
- 2:20 pm • Sprayed glass coated slide with monomer (ENB). Solvation occurred to disrupt smoothness of catalyst evenly applied.

Monomer coating solvated and pooled catalyst holes are clear, while cut is purple.



catalyst coating evenly placed on slide glass

- Subsequent spraying of monomer done to build thickness. Razor blade at edge indicates peelable coating. Allowed to dry fully before storing and labelling as RW8060-58-6 and -7 in ziplock bags.
- 3:00 pm • Made another catalyst solution 0.75 gm in 10 ml MeCl₂.
- We have been cleaning the flashing on each sample so far. This means that we have coated it as well.
- After wiping surface of RW8060-58-4 sample with acetone I attempted to weigh to help determine coating weight, but couldn't get accurate reading. The 6"x6" with flashing is too large to weigh with draft shield in place and air currents in lab-5 are too great to get accurate weight. Veeva tester may be used to determine thicknesses.

To Page No. 60

Witnessed & Understood by me,

James B Negey

Date

~~James B Negey~~

Invented by

R corded by

Russell Wells

Date

~~James B Negey~~

TITLE

From Page No.

Catalyst Activity Study Using Tricyclohexylphosphine

Materials: Tricyclohexylphosphine (TCHP, 97% from Aldrich),
ENB (CK8148-15-1), Grubbs's Catalyst (Boulder)

Procedure:

- 1) Polymerization with ENB and the Grubbs's catalyst is very fast. TCHP was added to the catalyst in order to slow down the polymerization. Different amounts of TCHP was added to the catalyst and then the ENB syringed into the mixture. Time of polymerization was then recorded. Table of results shown below:

M/I Ratio	TCHP/I Ratio	ENB (ml)	I (gms)	TCHP (gms)	Polymerization Time
5,000:1	2:1	3.0	0.0037	0.0025	1 min.
5,000:1	5:1	3.0	0.0037	0.0063	2 min.
5,000:1	10:1	3.0	0.0037	0.0125	4 min.
5,000:1	Control	3.0	0.0037	—	13 sec.
10,000:1	2:1	3.0	0.0018	0.0013	1 min. 4 sec.
10,000:1	5:1	3.0	0.0018	0.0031	2 min. 15 sec.
10,000:1	10:1	3.0	0.0018	0.0063	4 min. 50 sec.
10,000:1	Control	3.0	0.0018	—	22 sec.

M = Monomer (ENB)

I = Initiator (Grubbs's Catalyst)

TCHP = Tricyclohexylphosphine

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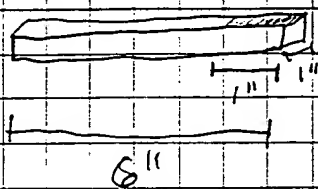
Date

Recorded by

Chris Yeck

From Page No. 50

CMP of polyolefins with
5,6-bis(chloromethyl)-2-norbornene



Polypropylene and LDPE strips were covered, as shown in the picture on the left, with $\sim 0.1\text{ mL}$ each of 5,6-bis(chloromethyl)-2-norbornene. The area covered was 1 in^2 at the end of a $6'' \times 1''$ strip. Two strips of each substrate type ~~or~~ were treated. A solution of 10 mg Grubbs' catalyst in $\sim 0.1\text{ mL}$ 5,6-bis(chloromethyl)-2-norbornene was prepared and quickly applied to one of each substrate types such that the solution was divided evenly between the two substrates. The catalyst soln was spread evenly over the 1 in^2 end of the substrate. The corresponding substrate without catalyst was inverted and placed in contact with the ~~XOR~~ catalyst solution. A 200 g weight was placed on the top of the substrates & the reaction was allowed to cure for 24 h .

To Page No. 51

Witnessed & Understood by me,

Amy C. Burke

Date

~~XXXXXX~~

Invented by

Jatin Kundell

Recorded by

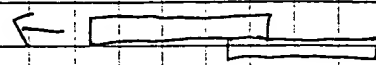
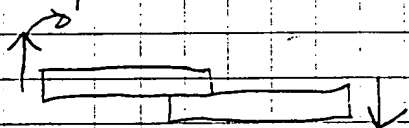
Jatin Kundell

Date

~~XXXXXX~~

From Page No. 50

After 24 h, The weight was removed from the samples & the bond strength was tested by hand.

When tested by shear:  The bond could not be broken by hand. When tested under peel conditions  After some

Slight initial effort, the samples peeled off with ease. That is, once a crack was formed it propagated at the adhesive-substrate interface with ease. The adhesive was a hard plastic-like material.

Patricia Kudell

~~SAO11~~

To Page N.

Witnessed & Understood by m,

Amey C. Burke

Date

~~SAO11~~

Invested by

Patricia Kudell

Recorded by

Patricia Kudell

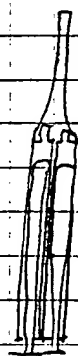
Date

~~SAO11~~

From Page No. _____

Bonding of Polypropylene Using 5,6-bis(chloromethyl)-2-norbornene, $WCl_4(O-\text{C}_6\text{H}_4)_2$, & SnBu₄ ~~delivered~~ as a 2-part adhesive from a cartridge.

A solution of 136mg (167μmol) $WCl_4(O-\text{C}_6\text{H}_4)_2$ in 1g 5,6-bis(chloromethyl)-2-norbornene + 40mg poly ENB (synthesized in 8290-p62 sample # 2) was prepared. A solution of 0.05mL SnBu₄ in 1g 5,6-bis(chloromethyl)-2-norbornene + 40mg poly ENB (8290, p62, sample #2) was prepared. The solutions were placed in A & B sides respectively of an in-house manufactured 1mL cartridge fashioned from 2 1mL ~~syringes~~ syringes (plastic/disposable) + a mixing chamber from the tip of a polypropylene pipette.



The mixing chamber was attached to the syringes with hot melt polyethylene adhesive.

To Page No. _____

Witnessed & Understood by me,

Date

Invent by

Date

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From Page N _____

The cartridge was used to deliver the adhesive to 6 sanded (w/ 100 grit paper) polypropylene coupons over a 1 in² area. The polypropylene coupons were mated with 6 additional sanded polypropylene coupons. The joints were held in place with a 170g weight.

After 24h, 3 samples were tested. Two pulled apart by hand & a third was pulled apart on the speedy tester with a stress at break of 57psi.

After 72h, the 3 remaining samples were tested. Two pulled apart by hand & the third was pulled apart on the speedy tester with a stress at break of 217psi. The samples that pulled apart by hand were not fully cured.

Jonathan Kudell

To Page No. _____

Witnessed & Understood by me.

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Dat

R corded by

*Florian Canelas**Jonathan Kudell**Jonathan Kudell*

From Page No. —

Bonding of Polypropylene Samples using ENB + low levels of Grubbs' catalyst

A solution of 0.5mg (0.6 μ mol) Grubbs catalyst in 1g (8.3mmol) 5-ethylidene-2-norbornene was prepared & quickly applied to one face of a 1" x 4" x 1/8" polypropylene substrate (sanded with 100grit paper & washed with acetone) over a 1in² area.

The coupons were mated with sanded polypropylene coupons such that a 1in² overlap ~~of the~~ overlap resulted. The solution was viscous within about 30 sec & the 5th coupon pair was joined after some viscosity had built up. The bonds were held in place with a 170g weight. Monomer: Catalyst ratio = 14000:1
Samples 8327-65-1, 2, 3, 4, 5.

The procedure ~~of the~~ above was repeated with 5 additional coupon pairs with the exception that

T Page N .

Witnessed & Understood by me,

Dorian Camelas

Date

~~_____~~

Invented by

D. K. Knoll

Date

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Recorded by

D. K. Knoll

From Page No. _____

0.25 mg (0.3 μ mol) Grubbs catalyst was dissolved in 1 g (8.3 mmol) 5-ethylidene-2-norbornene resulting in a monomer:catalyst ratio of 28000:1. As before, the last coupon (8327-66-10) was bonded after some viscosity had built up. Samples prepared were numbered 8327-66-6, 7, 8, 9, 10.

~~SAMPLES~~ Samples were pulled on the Instron ~~at a rate of~~ at 0.5 in/minute.

	stress @ max load
66-1	11.8 psi
66-2	12.7 psi
66-3	9.2 psi
66-4	fell apart
66-5	8.9 psi
ave	10.6 psi
std. dev	1.9 psi

	stress @ max load
66-6	30.2 psi
66-7	89.9 psi
66-8	63.6 psi
66-9	81.4 psi psi psi
66-10	196.8 psi
ave	92.4 psi
std. dev	62.7 psi

~~John Kruehll~~

To Page No. _____

Witnessed & Understood by m ,

Date

Invent d by

Date

Recorded by

Adrian Canelas

~~John Kruehll~~

John Kruehll

~~John Kruehll~~

From Page No. _____

The Effect of Applying ^{40%} ENB/60% NBD to a two part formulation. (5 + 10ml glass beads)

CONDITIONS

ENB	10.8g
NBD	25.2g
Europrene	6.35g
Cabosil	2.0g

PROCEDURE

ENB, NBD + Europrene were weighed into a glass jar and placed on a shaker for thirty minutes. 2g of Cabosil was added and shake for 5 minutes.

This was Side A of the formulation. It was transferred to a 10:1 Cartridge (The A-Side).

330mg of grubbs Catalyst was dissolved in 5g of ~~per~~ Dichloromethane (CH_2Cl_2) which was evaporated under flow of Nitrogen after 1.8g of Worthpar 100 processing oil was added. This resulted in a purple paste which was transferred to the B-Side of the Cartridge.

A 4" ~~static gun~~ ^{static gun} and mixing tip and static gun was used to dispense the formulation. (10ml glass beads)

OBSERVATION

fast curing seem to be a problem and the paste was too foamy. Also the mixing tip seem to be too long. Decided to do 50:50 ENB/NBD + reduce the mixing tip.

To Page No. _____

With ss d & Understood by me,

Date

Invented by

R corded by

Date

From Page No.

Repeat study of EMB/NBD to a two part formulation. (50:50) 5ml glass beads

CONDITIONS

EMB 18s
NBD 18s
Eureprene 6.35g
Cabosil 2g

PROCEDURE Same as 8363-64

RESULT 5mlSAMPLE 10ml

<u>SAMPLE ID</u>	<u>LOAD (psi)</u>	<u>SAMPLE ID</u>	<u>LOAD (psi)</u>	<u>SAMPLE ID</u>	<u>LOAD (psi)</u>
836364A-1	79.30	836364B-1	100.9	836365A-1	103.6
" - 2	114.50	" - 2	96.2	" - 2	137.0
" - 3	74.87	" - 3	58.9	" - 3	89.4
" - 4	98.24	" - 4	113.9	" - 4	99.1
" - 5	113.90	" - 5	101.2	" - 5	99.7
\bar{x} 96		\bar{x} 94		\bar{x} 106	
σ 19		σ 21		σ 18	

OBSERVATIONS

All of the samples failed adhesively

J. Taylor
~~Jonathan Taylor~~

To Page No.

Witnessed & Understood by me,

BQ

Date

~~8/13/78~~

Invented by

Recorded by

Jonathan Taylor

Date

~~8/13/78~~

From Page No. —

The Effects of Grubbs Catalyst with Cyclopentene/
Cyclohexene in a two part Adhesive formulation on
Polypropylene Substrate. (10:1 Cartridge)

FormulationsA-Side

	1A	2A
Norbornadiene	22.5g	22.5g
ENB	15.0g	15.0g
Blendex	7.5g	7.5g
Cabo-sil	1.0g	1.0g
Zeophores	4.0g	4.0g

B-Side

	1B	2B
Grubbs Catalyst	0.385g	0.385g
Cyclopentene	4.615g	—
Cyclohexene	—	3.765g
Cab-O-Sil	—	0.10g
Blendex	—	0.75g

PROCEDURE

A-Side formulation (1A+2A) were combined and mixed by hand in a plastic cup, but the blendex did not dissolve in the monomers (NBD/ENB). Europrene[®] was substituted for Blendex, which was weighed in a bottle with the monomers. It was placed on a roller overnight, the formulation was successfully done the following day. A total of 2.4g of Cabo-sil was used in the formulation.

The B-Sides were done separately.

OBSERVATIONS

The adhesive was found to be very fast cure, less than 15 seconds for 1B and less than 10 seconds for 2B. The 4" mixing tip had to be cut in half. We were able to get 6 samples for 1B and only 1 sample for 2B.

To Page N. —

Witnessed & Understood by me,

Shane McGowan

Date

~~_____~~

Invent d by

Recorded by

Jonathan Taylor

Date

~~_____~~

Proj ct N

B K N

8374

TITLE RESULTS

Fr m Page No. _____

SAMPLE ID	LOAD (PSI)	FAILURE MODE
83741B-1	165.68	Coh/Adh
" - 2	298.84	
" - 3	230.16	
" - 4	268.36	
" - 5	243.84	
" - 6	187.00	

7.232
8.45

SAMPLE ID	LOAD (PSI)	FAILURE MODE
8374112B-1	123.38	Coh/Adh

" $\frac{\pi}{2}$ - 2
" $\frac{\pi}{2}$ - 3

J. J. J. J.

To Page No. _____

Witnessed & Understood by me,

Dat

Inv nted by

Date

Rec rded by

Gonatan J. J.

From Page No.

Repeat of 8374-11 using $\frac{1}{10}$ the amount of Grubbs Catalyst on the B-Side. The A-Side stays the same.

<u>B-SIDE FORMULATION</u>	<u>1B</u>	<u>2B</u>	<u>A-SIDE</u>
Grubbs Catalyst	35mg 3.5mg	35mg	Same as 8374-11
Cyclopentene	4.9g	—	
Cyclohexene	—	4.0g	
Cal-O-Sil	0.20g	0.20g 0.02g	
Europrene		0.8g	

PROCEDURE Same as 8374-11

RESULT

<u>SAMPLE ID</u>	<u>LOAD (psi)</u>	<u>FAILURE MODE</u>
8374151B-1	—	
" - 2	4.20	Adh/coh
" - 3	4.90	
" - 4	7.90	
" - 5	5.46	
	\bar{x} 7	
	σ 2	

<u>SAMPLE ID</u>	<u>LOAD (psi)</u>	<u>FAILURE MODE</u>
8374152B-1	—	
" - 2	10.82	Adh/coh
" - 3	6.20	
" - 4	5.18	
" - 5	4.90	
	\bar{x} 7	
	σ 2	

To Page No.

Witnessed & Understood by me,

Shane Miller

Date

[Signature]

Invented by

Recorded by

Gina Rodriguez

Date

[Signature]

From Page No. _____

The Effect of Grubbs Catalyst with Cyclopentene, Cyclohexene in a two part Adhesive formulation on polypropylene substrate.

FORMULATION~~Norbornadiene~~A - SIDE

	<u>Amount</u>	<u>ACT Amount (x10)</u>
① Norbornadiene	22.5g	225g
② ENB	15.00g	150g
③ Europrene	7.5g	75g + 25g
④ Ca-bo-sil	1.0g	10g
⑤ Zeopheres	4.0g	40g

B - SIDE

	<u>1B</u>	<u>2B</u>	<u>1B</u>
Grubbs Catalyst	75mg	75mg	75mg
Cyclopentene	4.615g	—	2.308g
Cyclohexene	—	3.765g	2.308g
Ca-bo-sil	—	0.10g	0.10g
Europrene	—	0.75g	—

PROCEDURE Same as 8374-11OBSERVATION

Grubbs Catalyst and Cyclopentene cured in ~ 2 min resulting in a hard material. 50:50 Cyclopentene/Cyclohexene used instead.

T Pag No. _____

With ss d & Underst od by me,

Shane Mill

Dat

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Inv nted by

Recorded by

Gonathan Taylor

Date

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From Page No. _____

SAMPLE ID	LOAD (psi)	FAILURE MODE
837422B-1	12.42	Adh / Coh
" - 2	6.64	
" - 3	10.82	
" - 4	12.62	
" - 5	—	
	\bar{x} 11	
	σ 2	

SAMPLE ID	LOAD (psi)	FAILURE MODE
837422B-1	19.60	Adh / Coh
" - 2	5.20	
" - 3	11.62	
" - 4	9.10	
" - 5	—	
	\bar{x} 11	
	σ 5	

J. J. J.
~~6/12/80~~

To Page No. _____

Witnessed & Understood by me,

Shane Webb

Date

~~6/12/80~~

Investigated by

Recorded by

James J. J.

Date

~~6/12/80~~

From Page No.

Final
The Effect of Grubbs Catalyst with cyclohexene in
a two part adhesive formulation on polypropylene substrate.

A-Side #

Used 8374-22 (Masterbatch)

B-SIDECompoundAmount

Grubbs Catalyst

200mg

Cyclohexene

3.765g

Cabo-sil

0.20g

Europrene

0.75g

PROCEDURE

The cyclohexene and Europrene were weighed into
a small vial and placed on the paint shaker for 30 minutes.
The mixture was added to the Grubbs Catalyst in a plastic
cup, the Ca-bo-sil was added next and mixed well
by hand before transferring to the cartridge. Five samples
were made and cured overnight in the hood.

RESULT

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
832451-1	16hr	101.86	Adh
" - 2	"	56.64	"
" - 3	"	141.98	"
" - 4	"	180.00	"
" - 5	"	312.60	Adh / Coh
		$\bar{x} = 159$	
		$\sigma = 87$	

To Page No.

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Shane ML

Date

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Jonathan Taylor

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SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451B-1	15 min	163.52	Adh
" - 2	"	152.52	"
" - 3	"	160.24	"
" - 4	"	220.04	"
" - 5	"	169.58	"
		\bar{X} 173	
		σ 24	

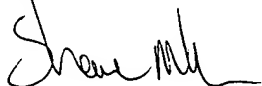
SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451C-1	30 min	193.08	Adh
" - 2	"	206.36	"
" - 3	"	212.22	"
" - 4	"	212.58	"
" - 5	"		
		\bar{X} 207	
		σ 9	

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451D-1	1 hr	230.84	Adh
" - 2	"	232.62	"
" - 3	"	257.92	"
" - 4	"	278.76	"
" - 5	"		
		\bar{X} 250	
		σ 20	

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451E-1	2 hr	197.52	Adh
" - 2	"	212.92	"
" - 3	"	238.80	"
" - 4	"	207.46	"
" - 5	"		
		\bar{X} 214	
		σ 15	

To Page No. _____

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Date

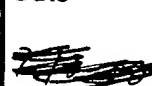


Invented by

Record d by



Date



From Page No. 52

The Effect of Grubbs Catalyst with Cyclohexene in a two part adhesive formulation on polypropylene substrate.

A-SIDE

Compound	Amount
80% Cyclohex methacrylate 20% Europrene	
Cyclohex methacrylate	18.24g
Europrene	4.56g
mono-2 (Methacryloyloxy)ethylsuccinate	1.45g

B-SIDE

Compound	Amount
Grubbs Catalyst	200mg
NaEtOBEt ₃	1.29g (used 1.25g)
Ca-bo-sil	0.20g
Cyclohexene	3.76g
Europrene	0.50g

PROCEDURE

The components were added together separately in a small plastic cup, hand mixed for ~ 2-5 minutes and then transferred to the cartridge. The Europrene and monomers were ^{weighed} placed into a small vial and placed on the paint shaker for 30 minutes. A stick gum and a 1/4" mixing tip was used to dispense the adhesive on 20 polypropylene samples.

OBSERVATION

These samples were left to cure over night. As they were still not cured after 2 hrs. they were pulled on the speeds tester after allowing to sit for 1 week.

Result Next page

To Page No. 54

Witnessed & Understood by me,

Shan Mh

Date

~~_____~~

Invented by

R c rded by

Jonathan Taylor

Date

~~_____~~

Proj ct N _____
B ok N 8374

TITLE

RESULT (CONT'D)

From Page No. _____

<u>RESULT</u>		
<u>SAMPLE ID</u>	<u>LOAD (psi)</u>	<u>FAILURE MODE</u>
837453-1	95.32	Col
" - 2	117.10	
" - 3	118.20	
" - 4	148.46	
" - 5	90.90	
" - 6	139.44	
" - 7	141.48	
" - 8	149.30	
" - 9	146.98	
" - 10	153.98	
" - 11	154.26	
" - 12	157.98	
" - 13	133.08	
" - 14	168.00	
" - 15	177.44	
" - 16	146.14	
" - 17	153.58	
" - 18	142.82	
" - 19	141.24	
	\bar{X} 140	
	σ 23	

J. Taylor
~~_____~~

To Page No. _____

Witnessed & Understood by me,

James M. [Signature]

Date

~~_____~~

Invited by

Record d by

Jonathan Taylor

Date

~~_____~~

From Pag No. _____

7-14-00

The effect of Grubbs Catalyst with Cyclohexene in a two part adhesive formulation on polypropylene Substrate.

A-SIDE

Used 8374-22 (masterbatch)

B-SIDE

Compound	Amount
Grubbs Catalyst	200 mg
Cyclohexene	3.765 g
Ca-bo-sil	0.20 g
Europrene	0.75 g

PROCEDURE

Same as in 8374-51

RESULT

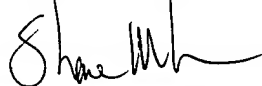
SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837455A-1	5 min	277.62	Adh / Coh
- 2		304.52	"
- 3		264.64	"
- 4		382.14	"
- 5		297.40	"
		\bar{x} 305	
		σ	

} samples were pulled between 5-10 minutes

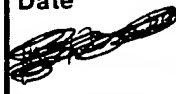
Sample ID	TIME	LOAD (psi)	FAILURE MODE
837455B-1	10 min	147.90	Adh
- 2		198.68	"
- 3		492.56	Coh
- 4		225.66	Adh
- 5		270.40	"
		\bar{x} 279	
		σ 120	

To Pag No. _____

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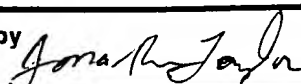


Date

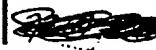


Invented by

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Date



From Page N. _____

Repeated 8374-50 at 5 minutes.

Sample ID	TIME	LOAD (psi)	FAILURE MODE
837450C-1	5 min	112.3	Adh
" - 2	"	315.2	Adh/Coh
" - 3	"	113.4	Adh
" - 4	"	151.2	"
" - 5	"	362.1	Adh/Coh
		\bar{X} 211	
		σ 106	

J. Taylor

To Page No. _____

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Date

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Jonathan Taylor

Date

From Page No.

~~11/1/84~~

The effect of Grubbs Catalyst with Cyclopentene.

A-SIDE

Used 8374-22 (masterbatch)

B-SIDE

Compound

Amount

A B

Grubbs Catalyst	200mg	140mg
Cyclopentene	4.8g	4.86g

procedure Same as in 8374-51

RESULT

SAMPLE ID	Time	LOAD (psi)	FAILURE MODE
837459A-1	1hr	81.50	Adh
" - 2	"	79.50	"
" - 3	"	98.34	"
" - 4	"	120.54	"
" - 5	"	63.66	"
		\bar{x} 89	
		σ 19	

Samples were pulled at 1hr-1:15

SAMPLE ID	Time	LOAD (psi)	FAILURE MODE
837459B-1	1hr	13.00	Adh
" - 2	"	18.20	"
" - 3	"	73.62	"
" - 4	"	72.14	"
" - 5	"	38.92	"
		\bar{x} 43	
		σ 26	

To Page No.

Witnessed & Understood by me,

Shane M

Date

[Signature]

Inv nted by

R c rded by

Jonathan Taylor

Date

[Signature]

From Page N

The Effect of Grubbs Catalyst with Cyclohexene on B-Side and ENB on A-Side. ~~8374/60~~

FORMULATIONA-SIDE

Compound	Amount
ENB	37.5g
Europrene	7.5g
Zeospheres	4.0g
Ca-bo-sil	1.0g

B-SIDE

Compound	Amount
Grubbs Catalyst	200mg
Cyclohexene	3.765g
Ca-bo-sil	0.2g
Europrene	0.75g

A-Side formulation was prepared by adding the ENB and Europrene together in a 4oz glass jar and placed on the paint shaker; then the fillers were added. The same was done on the B-Side.

RESULT

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837460-1	1.15 min	81.08	Adh
" - 2	"	57.04	"
" - 3	"	82.54	"
" - 4	"	87.10	"
" - 5	"	53.52	"

 \bar{x} 72 σ 14

J. Taylor
~~8374~~

To Page No.

Witnessed & Understood by me,

Shane M

Date



Invented by

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Jonathan Taylor

Date



From Page No. —

Super

The Effect of ^{Super} Grubbs Catalyst with Cyclohexene in a two part adhesive formulation on polypropylene substrate.

A-Side

used Pomp Masterbatch 8374-22

Compound	Amount	B-Side's
	8374-89A	8374-89B
^{Super} Grubbs Catalyst	100mg	50mg
Cyclohexene	3.75g	3.8g
Ca-bo-Sil	0.20g	0.20g

* A.II = 11, 20 & 30 minutes study

PROCEDURES

Both B-Sides were mixed separately by hand in a small plastic beaker and then transferred to the Cartridge. Five Samples were made for each set. They were allowed to cure over the week end.

Time study was done with 8374-89A.

Result

The 5th
Time (min)

Sample ID	Time (min)	Load (psi)	Failure Mode	Comment
837489A II 1	"	16.20	Adh	
" AII 2	"	392.14	Coh	Completely Cured in 15 min
" - 3	"	466.66	Adh/Coh	" "
" - 4	"	11.80	Adh	
" - 5	"	12.82	"	
		\bar{x} 180		
		σ 205		

J. Taylor
~~8374-22~~

To Page No. —

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Date

Invented by

Date

Record d by

Jonathan J. Taylor

From Page No. _____

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE	COMMENT
837489A-1	20min	15.74	Adh	
" - 2	"	11.92	"	
" - 3	"	446.24	Adh / Coh	Completely Cured in < 5min
" - 4	"	454.40	"	"
" - 5	"	474.24	"	"

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE	COMMENT
837489A-1	30min	385.96	Adh / Coh	Completely Cured in < 5min
" - 2	"	465.88	"	"
" - 3	"	465.10	"	"
" - 4	"	422.02	"	"
" - 5	"	\bar{x} 432		
		σ 81		

SAMPLE ID	TIME (hr)	LOAD (psi)	FAILURE MODE	COMMENT
837489A-1	48+	43.38	Adh	
" - 2	"	54.86	"	
" - 3	"	229.62	Coh / Adh	Completely Cured in < 5min
" - 4	"	41.40	Adh	
" - 5	"	42.70	"	
		\bar{x} 82		
		σ 74		

SAMPLE ID	TIME (hr)	LOAD (psi)	FAILURE	COMMENT
837489B-1	48+	—	—	
" - 2	"	18.26	Adh	
" - 3	"	35.92	"	
" - 4	"	28.02	"	
" - 5	"	46.68	"	
		\bar{x} 32		
		σ 10		

To Page N _____

Witnessed & Understood by me,

Share M

Date

~~Share M~~

Invented by

Recorded by

Jonathan Taylor

Date

~~Share M~~